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Misfit between sediment toxicity and chemistry

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Abstract—In the United States, the EPA EMAP-Estuaries Program and the NOAA Bioeffects surveys provide large data sets with which to test proposed relationships between sediment chemistry and toxicity. The conclusion is that no chemical measurement reliably predicts sediment toxicity. These findings challenge the use of chemical data to imply biological hazard and the way toxicity data are used in sediment management.

Introduction

There is a wealth of data on chemical concentrations in sediments of the coastal Unites States. The U. S. EPA (1997) estimate of the severity of sediment contamination in the United States used chemical concentrations measured at over 21,000 sampling stations. Among those 21,000 were 13,000 compiled by Daskalakis and O'Connor (1995) for just the coastal marine portion of the nation. After accounting for natural factors that affect concentrations, these data can become information on sediment contamination. It would be more satisfying and useful, however, if the chemical data could be converted to information on biological hazard. Within the United States, sediment quality guidelines (SQGs), proposed methods for doing just that, fall into two categories; those basing estimates of toxicity on water quality criteria and the assumption of equilibrium between sediment and pore water, and those based on compilations of simultaneously measured biological characteristics and bulk chemical concentrations. Here we demonstrate the shortcomings of SQGs and warn that except possibly in cases of extreme contamination, chemical data, by themselves, should not be used to predict hazards.

Definition of toxicity

Examining how well the various SQGs actually predict toxicity requires a definition of toxicity. Determinations based on laboratory tests depend on which species is tested, which response is measured, and the method of exposure. The many combinations among these choices make declarations of toxicity subjective but measuring survival of amphipods exposed to whole sediment for ten days has become a *de facto* standard. This test was introduced by Swartz et al. (1979) because surveys from a variety of locations showed that amphipod species decreased in abundance near sources of contamination.

In 1991 the amphipod test was added to the protocols for testing dredged material in the U. S. (U. S. EPA and U. S. Army, 1991). Prior to that sediment toxicity tests were conducted with deposit feeding polychaetes and suspension feeding mollusks. Evidence of the increased sensitivity of the amphipod test is that the frequency of toxic "hits" for dredged sediment near New York city has increased from less than 1% to greater than 30% (Del Vicario, 1998).

Since the introduction of the amphipod test, other tests have been proposed and used for assessing environmental quality. In the NOAA series of Bioeffect Surveys (Wolfe et al., 1994; Long et al., 1994, 1995a, 1996a, 1997) the whole-sediment 10-day amphipod survival test has always been used. Also used has been a test based on bacterial bioluminescence in dichloromethane extracts of homogenized sediment (after replacement of dichloromethane with the standard Microtox® diluent). In all surveys but one, a test of pore water toxicity has been used whereby sea urchin fertilization and embryo development are measured in water pressure-extracted from sediment. In the Long Island Sound (Wolfe et al., 1994) and Hudson Raritan Estuary (Long et al., 1995a) surveys, clam embryo survival and development were measured in elutriates (i.e. seawater extracts) of homogenized sediment. Among the newer tests (not used in Bioeffects Surveys) are those measuring growth of sand dollars and polychaetes (Rice et al., 1995) exposed to whole sediment. In general, all these tests yield a toxic response more frequently than the amphipod survival test.

Using all the Bioeffects Survey data, Long et al. (1996b) reported that *in toto* about 11% of the area within 22 U. S. estuaries was toxic by the amphipod test. However, with sediment deemed toxic on the basis of that test or the bacterial bioluminescence test or the sea-urchin test, they found that 91% of the estuarine area was covered by toxic sediments. Letting any test be used can put almost all sediments into the toxic category and nullify any effort to find a chemical (or any other) characteristic that predicts toxicity. On the other hand, relying on the tests that predate the amphipod test would find toxicity too infrequently to make the search for predictive characteristics even relevant. In this paper, sediment is considered toxic if there was less then 80% survival of amphipods during 10-day exposures to whole sediment (Long et al., 1996b, Macauley et al., 1996, and Strobel et al., 1995)

Theoretical limitations to chemical predictors

One set of SQGs are the Sediment Quality Criteria (SQC) for nonionic organic compounds (U. S. EPA, 1993). These are based on equilibrium partitioning theory and assume that pore water is in equilibrium with sediment and that, to be nontoxic, pore water must meet water quality standards. The equilibrium assumption allows pore water concentrations to be calculated from the more readily measured bulk sediment concentrations of nonionic compounds and total organic carbon. There are two questionable assumptions: 1) equilibrium exists and, 2) chemicals insufficiently soluble to exhibit aqueous phase toxicity cannot be toxic in sediment.

It is difficult to measure concentrations of organic compounds in small volumes of water separated from sediment. There are, therefore, few examples from the field where nonionic organic compound concentrations are reported for both the bulk sediment and pore water phases. The only actual measurements of pore water concentrations brought to bear by the U. S. EPA (1993) on the issue of demonstrating equilibrium are laboratory data with spiked systems. Unlike spiked sediments in a laboratory, particle-bound nonionic organic compounds in nature may not even be on the surface of particles. In nature, the particles go through all sorts of changes as they go into and out of organisms and as they encounter other particles and different levels of dissolved chemicals. The chemicals of interest can get covered under coatings of new additions to the system. While there is a tendency for the system to always shift in the direction of equilibrium and while that state is readily predicted by the SQC theory, actually reaching equilibrium is quite another matter. Soot is a primary source of polycyclic aromatic hydrocarbons (PAHs) that are ubiquitous nonionic organic contaminants in sediments. It has been convincingly shown that soot is definitely not in equilibrium with pore water (McGroody and Farrington, 1995 and McGroody et. al., 1996)

Equilibrium does not exist if either the kinetics of the system are too slow or the continual flow of mass and energy into a system counteracts the tendency toward equilibrium. Non-equilibrium is very common in the environment. No one would argue, for example, that overlying water in an estuary is in equilibrium with underlying sediment. There are great ranges over small horizontal distances in concentration of those compounds in surface sediments, even on a carbon-normalized basis.

A separate theoretical problem is that SQCs for nonionic organics compare calculated interstitial water concentrations with the Final Chronic Values (FCV) for aquatic toxicity. Compounds such as 2,4,7,8-tetrachlorodibenzo-p-dioxin, benzo(a)pyrene, higher chlorinated PCBs, and others with octanol/water coefficients greater than about 6, are insufficiently soluble to be toxic in the aqueous phase. Nonetheless, once ingested from food or sediment they exert biochemical changes that can lead to toxic effects.

Swartz et al. (1995) introduced a variation on the SQC for nonionic organics. This applies only to PAH compounds, adds up fractional toxicity attributable to each individual PAH, and does not require that each compound be sufficiently soluble to be toxic on its own. They calculated toxicity units for each of 13 PAH compounds as the ratio of their pore water concentration to their 10-day LC50 to amphipods. A sediment sample was predicted to be toxic if the sum of 13 toxicity units was greater than one. This SQC assumes that PAH compounds act additively and is based on the same equilibrium assumptions just described to calculate pore water concentrations. This version of the SQC is not limited by compound solubility. Unknown LC50s were calculated on the basis of relationships between toxicity and octanol/water coefficients and extended to compounds insufficiently soluble to actually have an LC50. Those compounds could still contribute fractional toxicity units.

For trace metals with sulfides that are less soluble than ferrous sulfide, there are SQGs based on acid-volatile sulfide (AVS) (DiToro et al., 1992). Sediments are predicted not to be toxic due to sulfide-insoluble metals whenever AVS exceeds simultaneously extracted metals (SEM). The theoretical problem is that such sediments are anoxic and therefore toxic. One could argue that equilibrium considerations do not apply and that, even in the presence of AVS, sufficient oxygen is available for aerobic life. However, that would nullify the equilibrium assumption at the core of the SQC for nonionic organic compounds. All equilibrium based SQGs assume that pore water must meet water quality criteria. Actually, except for very near the sediment/water interface, pore water in most coastal sediments is anoxic. This is not a lethal condition to benthic organisms because they develop strategies to obtain their oxygen and water-borne nourishment from the overlying water or they irrigate their environment with such water. Benthic organisms do not equilibrate with the pore water despite that being a key assumption of these SQGs.

The alternative to SQGs based on equilibrium partitioning are those based on bulk chemical concentrations. They are derived from collections of data from studies where chemical concentrations were measured along with some measure of biological response. Responses could be results from various bioassays with myriad species, benthic community changes, or various sediment quality guidelines from various sources. Data are subsequently sorted on the basis of bulk concentration and, in the case of Long et al. (1995b), the 50th and 10th percentile concentrations are designated the Effects Range Median (ERM) and Effect Range Low (ERL), respectively (Table 1). The ERMs are concentrations above which biological effects are expected frequently and ERLs are concentrations below which effects are expected to be rare. All of this is strictly empirical. A given data entry for copper, for example, has a biological response and a copper concentration but no evidence that copper, rather than some other sediment characteristic, was responsible for the response.

Long et al. (1998) introduced ERM Quotients, a calculation that emphasizes sediments that have many ERM exceedances or a few extreme exceedances. The idea is to divide the bulk concentration of each chemical by its ERM, add up all the ratios (there could be 25 if all chemicals in Table 1 were measured), and divide by the number of ratios. If the resulting ERM Quotient for a sediment sample is greater than one, it is claimed that there is an even greater chance of its being toxic than if there were simply just an ERM exceedance.

Empirical limitations to chemical predictors

With a 1500-sample data set created by combining results from the EPA and NOAA programs O'Connor et al. (1998) evaluated the ability of the various sediment quality criteria and guidelines to reliably predict sediment toxicity. Data were from the EPA EMAP-Estuaries program designed to randomly sample estuaries within entire biogeographical provinces of the United States. In particular, the three provinces sampled have been the Virginian Province (Chesapeake Bay, VA to Cape Cod, MA), the Louisianian Province (Gulf of Mexico except south of Tampa Bay, FL), the Carolinian Province (Cape Henry, VA to St. Lucie Inlet, FL). Because the samples were selected with a probability based design, there was no tendency or bias for them to be from particularly contaminated areas near harbors, industrial waterways, or other urban centers. Sediment samples from the NOAA Bioeffect Surveys, on the other hand, are from intensive sampling in urban bays. In general, therefore, they have higher concentrations of contaminants than the EMAP-E samples. For each sediment sample, in all cases, both bulk sediment concentrations and amphipod toxicity data are available.

The O'Connor et al. (1998) tabulation is repeated here in Table 2, increased with about 1000 samples from NOAA programs in the South Carolina, Georgia, and Florida, the 1995-1997 data from the EMAP Carolinian Province, the EPA Regional EMAP program in the New York/New Jersey Harbor system, and State of California/EPA/ NOAA programs in Southern California Bays and Estuaries and in San Diego Bay. Additional data have not changed the conclusions in any important way. Originally O'Connor et al. (1998) showed that only 38% of the samples with an ERM exceedance were actually toxic. With additional data, that percentage becomes 41%.

The other SQGs were so infrequently exceeded that they have not been included in Table 2. Nineteen of the 976 additional samples had concentrations that exceeded SQC for nonionic organics (8 were toxic) . So, the total for all 2475 samples becomes only 31 (13 of which were toxic) continuing to indicate the general inapplicability of that SQC. Similarly, there were only 17 samples (9 toxic) where the sum of toxic units was more than one based on the Swartz et al. (1995) SQC for 13 PAHs. There were 59 cases (37 toxic) where the ERM Quotient exceeded one

Acid volatile sulfide (AVS) and total metal (TotM) concentrations were measured in 1833 of the samples in Table 2. Since TotM, measured after digesting samples in concentrated HF, is greater than SEM, any sample with a TotM/AVS ratio less than one will also have an SEM/AVS ratio of less than one and be predicted to have no toxicity due to metals. Among the 1256 samples with TotM/AVS<1, only 139 (11%) were toxic. It is worth noting that the median Total Metal concentrations among samples with and without excess AVS were 1.63 and 1.65 µM/g, respectively, or essentially identical. The corresponding median AVS concentrations were very different, 6.44 and 0.43 µM/g, respectively. This guideline is very sensitive to AVS and leads to the conclusion that managing trace metal toxicity would require adjusting AVS while having little to do with metal contamination.

Within the 2475-sample data set, 2087 of the samples were not toxic. Since even a random choice would correctly identify nontoxic samples with an 84% frequency, the two tests that predict nontoxicity, SEM/AVS<1 and no ERL exceedances, were hardly challenged. As it is, 95% of the samples without an ERL exceedance (Table 2) came from among the nontoxic samples. The AVS-based SQG applies for the 1256 samples with TotM/AVS<1 and, as just stated, 89% of those came from the set of nontoxic samples.

Management implications

Guidelines that predict nontoxicity on the basis of chemical data were not seriously tested. Predictions of toxicity based on equilibrium partitioning for five nonionic compounds were few (31) and usually wrong (18 nontoxic). Predictions based on sums of toxic units for 13 PAH compounds were fewer still (17) and, again, often wrong (8 nontoxic). Predictions based on ERM Quotients were more common (59) but still rare and often wrong (22 nontoxic). Predictions based on ERM exceedances were the most common (453) and about as frequent as actual toxicity (388). Unfortunately only 41% of the ERM exceedances coincided with toxicity. Most (59%) of the sediments with chemical concentrations high enough to exceed an ERM were nontoxic.

There is no obvious way to fix this misfit between chemical and toxicity data. Requiring a more than 20% loss of amphipods, or using a less sensitive assay, or requiring a "hit" in more than one assay would all result in fewer samples being declared toxic. However, all the various SQG exceedances would still apply and their corresponding frequencies of being "correct" would be even lower. To go the other way and use a more sensitive assay would increase the frequency of "correct" predictions but also increase the percentage of samples that are toxic but not predicted to be so by any SQG (already 202, or 52%, of the 388 toxic samples in Table 2 do not exceed any chemical SQG). In the extreme, almost all samples could be declared toxic if any test could be brought to bear (example, the 91% spatial extent of toxicity of Long et al. (1996b). In that case predictions based on chemistry or on anything else would be useless.

Though environmental agencies throughout North America (Long et al., 1998) do so, SQGs should not be used to attach biological meaning to chemical data. One large scale example is the U. S. EPA (1997) report on the incidence of contaminated sediments in the United States, where "contaminated" sediments are said to be "toxic" primarily on the basis of SQGs (in particular ERMs).

On the other hand, because chemical measurements do not indicate toxicity, bioassays are used in the U. S. to determine if dredged sediment can be dumped at sea. Since the connection between toxicity and chemistry is weak, this could imply that controls on chemical inputs to harbors may have little or no influence on whether harbor sediments can be one day dredged and dumped at sea. It is always possible that toxicity is due to unmeasured chemicals. If so, it would be futile to control inputs of known chemicals.

The underlying dilemma is that sediment management decisions put so much emphasis on SQGs or toxicity tests. The former are not very predictive of biological hazard and the latter are somewhat arbitrary in the sense that determinations of "toxicity" depend on the test applied. Many aspects of the sediment could come into consideration. In addition to chemical and toxicity data, thoughtful consideration should be given to the consequence of placing toxic sediment at an offshore disposal site relative to not dredging at all or of putting the material on land. Sediment toxic to amphipods is by no means toxic to all forms of life and even sediments that fail the amphipod bioassay sometimes support amphipods *in situ* (Strobel et al., 1995, Macauley et al., 1996). Lastly, in assessing risks to natural resources and to humans, the location and spatial scale of contaminated sediments should be considered at least as important as their chemical characteristics and behavior in bioassays.

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Table 1. ER-Ls and ER-Ms from Long et al. (1995b). ER-Ls are bulk sediment concentrations (dry-weight) below which sediment is unlikely to be toxic. ER-Ms are concentrations above which toxicity is probable.

Chem.	ERL		ERM	
Ag	1	ppm	3.7	ppm
As	8.2		70	
Cd	1.2		9.6	
Cr	81		370	
Cu	34		270	
Hg	0.15		0.71	
Ni	21		52	
Pb	47		220	
Zn	150		410	
p,p'-DDE	2.2	ppb	27	ppb
Total DDT[a]	1.6		46	
Total PCB	23		180	
LMWPAH[a]	550		3160	
HWWPAH[a]	1700		9600	
Total PAH[a]	4000		45000	
Acenapthene	16		500	
Acenaphthylene	44		640	
Anthracene	85		1100	
Flourene	19		540	
2-Methylnaphthalene	70		670	
Naphthalene	160		2100	
Phenanthrene	240		1500	
Benzo(a)pyrene	261		1600	
Chrysene	384		2800	
Dibenzo(a,h)anthracene	63		260	
Fluoranthene	600		5100	
Pyrene	670		2600	

[a]DDE is part of the Total DDT and individual PAH compounds are part of the Total PAH, LMWPAH (2- and 3-ring compounds), and HMWPAH (more tan 3-ring compounds). However, double and triple counting is of no consequence when we are interested in knowing only whether there is at least one exceedance of ERL or ERM.

Table 2. Total numbers of samples per data set, number of ER-L non-exceedances, number of ER-L non-exceedances that were toxic to amphipods, number with at least one ER-M exceedance, number with at least one ER-M exceedance that were toxic, and total number that were toxic (<80% survival after ten days). Sources are EMAPV (EMAP Virginian Province, 1990-93 data), EMAPL (EMAP Louisianian Province, 1991-94 data), EMAPC (EMAP Carolinian Province, 1994-97 data), REMAP (Regional EMAP in NY/NJ Harbor System), LIS (Bioeffect Survey in Long Island Sound), HRE (Bioeffect Survey in Hudson-Raritan Estuary), BOS (Bioeffect Survey in Boston Harbor), LA (Bioeffect Survey in Los Angeles Harbor), TB (Bioeffect Survey in Tampa Bay), SC/GA (South Carolina and Georgia), WestFL (Western Florida Bays), BisBay (Biscayne Bay), CAEst (Southern California Estuaries), and SDBay (San Diego Bay)

Data set	Total samples	Total samples w/ all<ERL	Toxic samples w/ all<ERL	Total samples w/ at least one >ERM	Toxic samples w/ at least one>ERM	Total toxic smpls(%)
EMAPV	537	211	12	82	25	59 [11]
EMAPL	642	195	9	11	3	68 [11]
EMAPC	194	127	3	13	0	3 [2]
REMAP	168	46	0	66	18	24 [14]
LIS	63	2	2	21	13	32 [51]
HRE	38	2	0	34	18	20 [53]
BOS	30	0	0	22	4	6 [20]
LA	45	0	0	40	14	15 [33]
TB	61	5	1	25	9	10 [16]
SC/GA	144	37	0	10	1	2 [1]
WestFL	71	11	0	31	1	1 [1]
BisBay	226	90	6	35	18	25 [11]
CAest	43	0	0	24	16	23 [53]
SDBay	213	4	0	105	64	124 [58]
TOTAL	2475	730	33	453	186	388 [16]

Sources;EMAPV (Strobel et al., 1995), EMAPL (Macauley et al., 1996), EMAPC (Hyland et al, 1996, 1998, and pers. comm.), REMAP (Adams et al., 1998), LIS (Wolfe et al., 1994), HRE (Long et al., 1995b), BOS (Long et al., 1996a), LA (Sapudar et al., 1994), TB (Long et al., 1994), SC/GA (Long et al., 1998), WestFL (Long et al., 1997), BisBAY (Long et al., in press), CAest (Anderson et al., 1997), SDBay (Fairey, 1996)